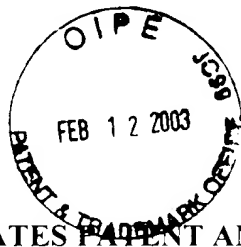


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TC 1700

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF: :

Mario SANDOR, et al. :

SERIAL NO.: 09/702,724 :

FILED: NOVEMBER 1, 2000 :

FOR: AQUEOUS POLYMER DISPERSIONS

DECLARATION UNDER 37 C.F.R. §1.132

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Now comes Dr. Cheng-Le Zhao who deposes and states:

1. I am a graduate of University of Strasbourg, France, and received my doctorate degree in the year 1987.
2. I have been employed by BASF for 14 years as a Research Associate in the field of aqueous polymer dispersions.
3. I am one of the inventors of U.S. application, Ser. No. 09/702,724.
4. I have read and fully understood the Office Actions of May 23, 2002, and August 27, 2002 and the prior art cited therein.
5. The following experiments were carried out by me or under my direct supervision.

5.1 Experiments

In order to show the superior properties of the claimed polymer lattices I have prepared polymer lattices D1, D2 and D3 according to the present invention and the comparative lattices V1, V2 and V3 by the processes described below.

General polymerization recipe:

A polymerization reactor was charged with 352.94 ml of water and 36.91 g of a polystyrene seed latex (32.1 % by weight; average particle diameter $d_{50} = 30$ nm) and this initial charge was heated to 90°C. Then 2 g of initiator solution were added in one portion. Commencing simultaneously, the remaining initiator solution was added over the course of 165 minutes and the monomer emulsion I was added over the course of 120 minutes to the polymerization reactor while maintaining the temperature. After 120 minutes, calculated from the beginning of the monomer addition, the monomer emulsion II was started and added over the course of 45 minutes to the polymerization reactor. After the end of the additions of monomer and initiator, the 85°C were maintained for a further 30 minutes and then the mixture was cooled to 75 °C and 7.76 g of 29 % strength by weight aqueous ammonia were added. To further reduce the amount of residual monomers, the following chemical stripping process was applied: 11.25 g of a 4% strength solution of t-butyl hydroperoxide in water and 11.27 g of a 4% strength solution of sodium acetone bisulfite solution in water were fed separately and continuously into the reactor over an one hour period. The mixture was subsequently cooled to room temperature and neutralized to a pH of 7.0 to 8.0 with 29 % strength by weight aqueous ammonia solution, and 3.3 g of a biocide were added. The composition of monomer emulsions I and II were as follows:

Monomer emulsion I:

343.50 g	of water
60.00 g	of emulsifier 1
15.00 g	of emulsifier 2
13.50 g	of methacrylic acid
x g	of tert.-dodecyl mercaptan
298.80 g	of n-butyl acrylate
380,70 g	of methyl methacrylates

Monomer emulsion II:

72.50 g	of water
30.00 g	of emulsifier 1
15.00 g	of emulsifier 2
9.00 g	of acrylic acid
y g	of tert.-dodecyl mercaptan
180.45 g	of methyl methacrylate
17.55 g	of a 25% strength by weight solution of N-(2'-methacryloyloxyethyl)imidazolin-2-one in methyl methacrylate

Emulsifier 1: A 15 % strength aqueous solution of sodium dodecyl sulfate under the trade name of Texapon K12-15 from Cognis Corporation, 300 Brookside Avenue, Ambler, Pennsylvania, USA;

Emulsifier 2: A 33 % strength aqueous solution of sodium lauryl ether sulfate, 30-EO moles under the trade name of Disponil FES 77 IS from Cognis Corporation, 300 Brookside Avenue, Ambler, Pennsylvania, USA;

The resulting dispersion were characterized with regard to coagulum, solids content, pH and average particle diameter, which was measured by means of photon correlation spectroscopy (d_{50} value). The results are given in following table:

	VD1	D1	D2	D3	V2	V3
x g	0	1.80	3.60	0	0.90	1.80
y g	0	0	0	1.80	0.90	1.80
Total Solids Content [%]	46.6	48.2	48.1	46.9	48.5	44.5
pH	7.8	8.0	7.3	7.2	7.9	7.8
Particle Size [nm]	131.4	110.9	139.0	133.2	127.0	92.2
Coagulum [%]	0.04	0.15	0.04	0.03	0.18	0.14

Each latex was formulated into a latex paint according the following paint formulation:

Constituent	Parts (g)
Propylene glycol	30.0
Water	10.0
Biocide ¹⁾	1.6
Methylcarbitol	16.0
Pigment dispersant ²⁾	15.0
Defoamer ³⁾	2.0
Thickener ⁴⁾	28.2
Pigment ⁵⁾	239.6

Grind 30 min. at 1000 RPM and then add the following constituents:

Dispersion (50%) [*]	525.6
Coalescence aid ⁶⁾	32.9
Ammonia (25%)	0.8
Water	89.4
Defoamer ⁷⁾	2.4

1) Kathon LX 1.5%, supplied by Rohm&Haas

2) Tamol 1124, supplied by Rohm & Haas

3) Drewplus L108, supplied by Drew Industrial Division of Ashland Specialty Chemical Co.

4) Optiflo L 100 supplied by Süd Chemie

5) Tipure R 706 Dry TiO₂ supplied by DuPont

6) Texanol, supplied by Eastman Chemical Products

7) Colloids 643, supplied by Rhodia

6. Results

The high gloss paints were tested with regard to block-resistance, block resistance and specular gloss @20° and @60° according to the test procedures outlined on page 24 of 09/702,724. The results for the paints obtained from dispersions D1, D2, D3 and V1 to V3 are summarized in the following table:

	V1	D1	D2	D3	V2	V3
Block Resistance						
B1	10	9-10	10	9	9	8-9
B2	10	10	9-10	10	10	9
B3	9	9	8	9	8	7-8
B4	10	10	9	10	10	8
Gloss						
@ 20°	46.2	47.2	47.9	44.3	40.3	46.2
@ 60°	77.9	78.0	79.0	76.3	73.8	77.7
Scrub Cycles	677	797	748	1140	1059	1089

B1 = Leneta sheet, 30 min, 50°C, 0.5 kg

B2 = Leneta sheet, 24 h, room temperature, 1 kg

B3 = Sealing paper, 30 min, 50°C, 0.5 kg

B4 = Sealing paper, 24 h, room temperature, 1 kg

As can be seen from the comparative results of the latices (D1 and D2, according to the present invention, versus V1), the use of a chain transfer agent in the 1st polymerisation stage results in paints which show increased gloss and have only slightly reduced block resistance. As can be seen from the comparative results of the three pairs of latices (D1, according to the present invention, versus V2, D2, according to the present invention, versus V3 and D3, according to the present invention versus V2), the use of a chain transfer agent in both stages at total levels equal to that used in the first stage (or in the second stage) result in detrimental effects on gloss. The use of a chain transfer agent in the 2nd polymerisation stage results in polymer latices showing reduced gloss compared to latices wherein the chain transfer agent is used in the 1st polymerisation stage (D1 versus D3). However, when the chain transfer agent is used in the 2nd polymerisation stage, the scrub resistance is higher than either the case where no chain transfer agent is used (V1) or the case where

the chain transfer agent is used in both stages (D3 versus V2 or V3). The higher scrub resistance associated with the use of a chain transfer agent in the 2nd stage can also be clearly seen from table 2 on page 25 of Ser. No. 09/702,724. Therefore the use of a chain transfer agent in either the 1st stage or the 2nd stage of the emulsion polymerisation, leads to better properties compared to those latices obtained by emulsion polymerisations without any chain transfer agent or with the chain transfer agent distributed in both stages.

7. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

8. Further deponent saith not.

Charlotte NC, Jan 27, 2003

Charles F. Hew